DOCKET NO: 264626US0PCT

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :

OLEG STENZEL, ET AL. : EXAMINER: HANOR, SERENA L.

SERIAL NO: 10/523,029 :

FILED: SEPTEMBER 7, 2005 : GROUP ART UNIT: 1793

FOR: HIGHLY DISPERSIBLE

PRECIPITATED SILICA

APPEAL BRIEF

COMMISSIONER FOR PATENTS ALEXANDRIA, VIRGINIA 22313

SIR:

This is an appeal of the Final Rejection dated March 10, 2009 of Claims 1-4 and 21-28. A Notice of Appeal is submitted herewith.

I. REAL PARTY IN INTEREST

The real party in interest in this appeal is Evonik Degussa GmbH, having an address at Rellinghauser Strasse 1-11, 45128 Essen, Germany.

II. RELATED APPEALS AND INTERFERENCES

Appellants, Appellants' legal representative and the assignee are aware of no appeals or interferences which will directly affect or be directly affected by or have a bearing on the Board's decision in this appeal. However, an appeal in US 10/522,672 may have a bearing on the Board's decision in this appeal.

III. STATUS OF THE CLAIMS

Claims 1-4 and 21-28 stand rejected and are herein appealed. Claims 5-20 stand withdrawn from consideration. Claims 19 and 20 have been canceled.

IV. STATUS OF THE AMENDMENTS

No amendment under 37 CFR 1.116 has been filed.

V. SUMMARY OF THE CLAIMED SUBJECT MATTER

A summary of the claimed subject matter on appeal, as claimed in independent Claim 1, is mapped out below, with reference to page and line numbers in the specification added in **[bold]** after each element.

A precipitated silica, [page 5, lines 3-4] wherein said silica has the following properties:

BET surface area	190 - 302 m^2/g , [page 5, lines 4 and 29]
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CTAB surface area $\geq 170 \text{ m}^2/\text{g}$, [page 5, line 5]

DBP number 200 - 300 g/(100 g), [page 5, lines 5-6]

and

Sears number V_2 10-20 ml/ (5 g). [page 5, lines 7-8]

VI. GROUNDS OF REJECTION

Ground (A)

Claims 1-4 and 11-28 stand rejected under 35 USC 103(a) as unpatentable over US 6,180,876 (<u>Uhrlandt et al</u>).

Ground (B)

Claims 1, 2, 4, and 21-25 stand rejected under 35 USC 103(a) as unpatentable over US 5,705,137 (Goerl et al).

Ground (C)

Claims 1, 2, and 21-25 stand rejected under 35 USC 103(a) as unpatentable over US 2003/0003040 (Lindner et al).

Ground (D)

Claims 1-4 stand provisionally rejected on grounds of obviousness-type double patenting over Claims 1 and 13 of <u>Uhrlandt et al.</u>

VII. ARGUMENT

Ground (A)

Claims 1-4 and 11-28 stand rejected under 35 USC 103(a) as unpatentable over Uhrlandt et al. That rejection is untenable and should not be sustained.

As recited in Claim 1, an embodiment of the present invention is a precipitated silica, wherein said silica has the following properties:

BET surface area $190 - 302 \text{ m}^2/\text{g}$,

CTAB surface area $\geq 170 \text{ m}^2/\text{g}$,

DBP number 200 - 300 g/(100 g), and

Sears number V_2 10-20 ml/ (5 g).

Uhrlandt et al discloses a precipitated silica having a BET surface area of 120 to 300 m^2/g , a CTAB surface area of 100 to 300 m^2/g , a Sears number of 6 to 25 ml, a DBP number of 150 to 300 g/100 g and a WK coefficient of < 3.4 (column 1, lines 40-50). Thus, in

general, these parameters are significantly broader and inclusive of and/or overlap with the corresponding parameters of the present claims. In addition, all of the Examples of <u>Uhrlandt</u> et al are outside the terms of the present claims.

The present invention is a selection invention from the relatively broader disclosures in the prior art, such as <u>Uhrlandt et al</u>, as now demonstrated. In other words, the present inventors have succeeded in finding and demarcating, from an unimaginable number of theoretically disclosed silicas in <u>Uhrlandt et al</u>, those which are suitable for incorporation in motor bikes, trucks and high-speed automobile tires. The fact that the selected silicas have a considerable technical advantage will be shown below.

As described in the specification at page 2, line 13 through page 4, line 16, silicas for commercial-vehicle tires have a requirements profile that is clearly different from that of silicas for passenger-car tires. This area of application imposes the following special requirements, among others, on the silica: enabling the manufacture of commercial-vehicle tires that, compared with passenger-car tires, have much higher mileage, less wear, off-road ability, good tensile strength and improved traction. On the other hand, silicas for normal passenger-car tires must guarantee low rolling resistance. In addition, the requirements for mileage are much less stringent.

<u>Uhrlandt et al</u>, on the other hand, is concerned with improved dispersability in rubber mixtures (column 1, lines 34- 36). Examples 10 and 11 of <u>Uhrlandt et al</u> provide detailed knowledge on which silicas can be used for incorporation in the running surface of a passenger-car tire and which can be used for incorporation in the carcass of a normal passenger-car tire.

Between the publication of <u>Uhrlandt et al</u> and the filing date of the present invention, it was found that silicas that are suitable for incorporation in normal passenger-car tires are not suitable for incorporation in commercial-vehicle tires, as discussed above. One could not

conclude as such from Uhrlandt et al.

As discussed above, <u>Uhrlandt et al</u> discloses very broad ranges for BET, CTAB, DBP number and Sears number. From this, it is statistically possible to calculate a very large number of theoretically disclosed parameter combinations. By comparison, if combination possibilities according to Claim 1 are considered, it is evident that the claimed "combination of parameters," in effect, claims only a very small and narrow part of the disclosure of <u>Uhrlandt et al</u>. It is therefore necessary to assess the combination of parameters, as opposed to each parameter separately.

Applicants wish to emphasize an important understanding that must be given to precipitated silicas that are to be used as fillers in the preparation of rubber materials, particularly fillers that are to be used in the construction of tire treads, that has long been known to those of skill in the art, and is described in US 5,929,156 (Fultz et al) at column 3, lines 46-54, as follows:

It is well known that a single physical characteristic, such as surface area or particle size, does little to describe a silica product or to predict is behavior in a specific application. The mechanisms which govern how a particular silica product performs in a given end-use can be extraordinarily complex and are often not well understood; thus, linking one or even a few conventionally-measured silica product physical properties to particular end-use performance characteristics is extremely difficult and potentially misleading.

One property which is particularly important for commercial vehicles is improved cut & chip and chunking resistance, and bar tear resistance, (all of which are a measure of high-temperature tear resistance), as described in the specification at page 9, line 18ff and page 18, first full paragraph. The effectiveness of such properties can be measured by the Die C test according to ASTM D 624, wherein the higher the value, the greater the high-temperature tear resistance. In a Declaration under 37 C.F.R. § 1.132 of André Wehmeier, filed June 20, 2008 (first Wehmeier Declaration), Die C for present Example 1 was 75 N/mm, while it was

52 N/mm and 55 N/mm for Examples 4 and 5, respectively, of <u>Uhrlandt et al.</u>

A Declaration under 37 C.F.R. § 1.132 of André Wehmeier, filed January 15, 2009 (second Wehmeier Declaration) declares that the closest prior art to the presently-claimed invention in <u>Uhrlandt et al</u> is Example 4 therein. Said Example 4 has both a BET surface area of 185 m²/g and a CTAB surface area of 163 m²/g, and both values lie outside of present Claim 1. Applicants have clearly shown in the first Wehmeier Declaration that an embodiment of the present invention, i.e., Example 1, has superior high-temperature tear resistance to Example 4 (and Example 5) of <u>Uhrlandt et al</u>. In addition, the second Wehmeier Declaration shows two additional examples within the terms of the present claims, i.e., Examples 1A and 1B, which together with the data in the first Wehmeier Declaration, establishes a trend that by operating within the full scope of the present invention, superior high-temperature tear resistance to <u>Uhrlandt et al</u> is established. Compare *In re Kollman*, 595 F.2d 48, 56, 201 USPQ 193, 199 (CCPA 1979).

In the Final Rejection the Examiner notes that the feature of superior hightemperature tear resistance of a tire is not recited in the claims, that limitations from the specification are not read into the claims (citation omitted), that the intended use of the precipitated silicas carries no patentable weight, and that there is no mention of tires in the claims, as bases for adhering to the rejection.

In reply, the property of superior high-temperature tear resistance that the presently-claimed silica imparts to a tire has been shown to be an inherent property of the silica and thus, need not be recited in the claims. Indeed, "[f]rom the standpoint of patent law, a compound and all its properties are inseparable." *In re Papesch*, 315 F2d 381, 391, 137 USPQ 43, 51 (CCPA 1963). Nor is it relevant that there is no mention of tires in the claims. For example, a new compound shown to unexpectedly treat cancer would be patentable whether or not a claim to the compound mentions cancer.

With regard to the second Wehmeier Declaration, the Examiner finds that the Median Die C values for Example 4 of <u>Uhrlandt et al</u> and Example 1A "are similar," and adds that the fact that the high-temperature tearing resistance for Examples 1A and 1B are higher than that of the silica of Example 4 of <u>Uhrlandt et al</u> "is irrelevant because, said property is not claimed."

In reply, aside from the fact that it is not clear how a property is claimed, the property is inherent, as discussed above. Nor is the trend established by Examples 1A and 1B similar to or suggested by Example 4 of <u>Uhrlandt et al.</u>

While the Examiner cites precedent to the effect that prior art is not limited to specific examples, precedent also holds that a *prima facie* case of obviousness can be overcome by demonstrating unexpected results over the closest prior art. Applicants have done so.

For all the above reasons, it is respectfully requested that the rejection over <u>Uhrlandt</u> et al be REVERSED.

Ground (B)

Claims 1, 2, 4, and 21-25 stand rejected under 35 USC 103(a) as unpatentable over Goerl et al. That rejection is untenable and should not be sustained.

Goerl et al discloses that it was known in the prior art that precipitated silicas are known that have, *inter alia*, a BET surface area of 35 to 350 m²/g, a silanol group density (V₂-NaOH consumption) [Sears number] of 6 to 20 ml, a CTAB surface area of 30 to 350 m²/g, and a DBP index of 150 to 300 ml/100g (column 2, lines 12-22). Goerl et al's invention requires, *inter alia*, a CTAB surface area of greater than 200 m²/g, and more particularly, precipitated silicas having a CTAB surface area of 200 to 400 m²/g, a DBP index of from 230 to 380 ml/100g, a Sears number of 20 to 30 ml (column 2, lines 33-38). However, like Uhrlandt et al above, Goerl et al does not disclose any particular silicas

meeting all the terms of the present claims. Indeed, all of the examples of <u>Goerl et al</u> have a Sears number of greater than 20, and are thus all outside the terms of the present claims.

Thus, <u>Goerl et al</u> is no more relevant than <u>Uhrlandt et al</u>, discussed above.

In addition, as Wehmeier declares in the second Wehmeier Declaration, Example 4 of Uhrlandt et al is the closest prior art. Indeed, the silicas of Uhrlandt et al are closer to the presently-claimed silicas, as can be easily ascertained by simply comparing the Sears numbers of Uhrlandt et al, which broadly encompass the Sears number range of the present claims, and Goerl et al, whose Sears number range of 20-30 only incidentally touches the presently-recited maximum of 20, and the examples and therefore preferred embodiments of, Goerl et al all have Sears numbers greater than 20. Thus, to the extent Applicants have demonstrated unexpected results over Uhrlandt et al, such results can be extrapolated to the less pertinent prior art of Goerl et al. Compare Ex parte Humber, 217 USPQ 265 (Bd. Pat. App. & Inter. 1981) (copy enclosed) (comparative data showing the claimed chlorine-containing compounds to be unexpected over various (non-prior art) chlorine-containing isomers was accepted as more probative over prior art, drawn to non-chlorine containing analogs of the claimed compounds, asserted to be closest.)

In the Final Rejection, the Examiner cites the same precedent as discussed above under Ground (A) that prior art is not limited to particular examples. Applicants' reply thereto is hereby incorporated by reference.

The Examiner further finds that it is unclear how the *Humber* case applies.

In reply, *Humber* is an example of a case in which an indirect comparison was held to be valid for demonstrating unexpected results. Since the evidence of record demonstrates unexpected results over <u>Uhrlandt et al</u>, and <u>Uhrlandt et al</u> is closer to the presently-claimed invention than <u>Goerl et al</u>, the evidence is sufficient to demonstrate patentability over <u>Goerl et al</u>.

For all the above reasons, it is respectfully requested that this rejection be REVERSED.

Ground (C)

Claims 1, 2, and 21-25 stand rejected under 35 USC 103(a) as unpatentable over Lindner et al. That rejection is untenable and should not be sustained.

Lindner et al discloses precipitated silicas having, *inter alia*, a BET surface area of from 50 to 700 m²/g, a DBP absorption of from 100 to 450g/100g, and a Sears number greater than 20, preferably greater than 25, and particular preference greater than 28 [0028]-[0033]. The present claims, on the other hand, require a maximum Sears number of 20.

As stated above with regard to the rejection over <u>Goerl et al</u>, Wehmeier declares in the second Wehmeier Declaration, Example 4 of <u>Uhrlandt et al</u> is the closest prior art. Indeed, the silicas of <u>Uhrlandt et al</u> are closer to the presently-claimed silicas, as can be easily ascertained by simply comparing the Sears numbers of <u>Uhrlandt et al</u>, which broadly encompass the Sears number range of the present claims, and <u>Lindner et al</u>, whose Sears number is greater than 20, preferably greater than 25, and particular preference greater than 28 [0028]-[0033]. Thus, to the extent Applicants have demonstrated unexpected results over <u>Uhrlandt et al</u>, such results can be extrapolated to the less pertinent prior art of <u>Lindner et al</u>. Compare *Ex parte Humber*, *supra*.

In the Final Rejection, the Examiner finds that Claim 4 of <u>Lindner et al</u> is evidence that a Sears number of 20 is contemplated. Applicants' reply is that taken as a whole, <u>Lindner et al</u> leads one of ordinary skill in the art to Sears numbers greater than 20.

In the Final Rejection, the Examiner's response to the above arguments is otherwise the same as the response to the arguments under Ground (B). Applicants' reply under Ground (B) is hereby incorporated by reference.

Application No. 10/523,029

Appeal Brief

For all the above reasons, it is respectfully requested that this rejection be

REVERSED.

Ground (D)

Claims 1-4 stand provisionally rejected on grounds of obviousness-type double

patenting over Claims 1 and 13 of <u>Uhrlandt et al</u>. That rejection is untenable and should not

be sustained.

These claims of <u>Uhrlandt et al</u> are no more relevant than the disclosure in <u>Uhrlandt et</u>

al, discussed above. Thus, the arguments in traversal of the rejection over Uhrlandt et alas

prior art is hereby incorporated by reference. Accordingly, it is respectfully requested that

this rejection be REVERSED.

VIII. CONCLUSION

For the above reasons, it is respectfully requested that the rejections be REVERSED.

Respectfully submitted,

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CLAIMS APPENDIX

Claim 1. A precipitated silica, wherein said silica has the following properties:

BET surface area $190 - 302 \text{ m}^2/\text{g}$,

CTAB surface area $\geq 170 \text{ m}^2/\text{g}$,

DBP number 200 - 300 g/(100 g), and

Sears number V_2 10-20 ml/ (5 g).

Claim 2. The precipitated silica as claimed in claim l, wherein the CTAB surface area is not more than $300 \text{ m}^2/\text{g}$.

Claim 3. The precipitated silica as claimed in claim 1, having a WK coefficient of \leq 3.4 (ratio of the peak height of the particles which cannot be broken down by ultrasound in the size range 1.0 - 100 μ m, to the peak height of the broken-down particles in the size range $<1.0~\mu$ m).

Claim 4. The precipitated silica as claimed in claim 1, wherein the surfaces of said silica have been modified with organosilanes of the formulae I to III:

$$[\operatorname{SiR}^1{}_n(\operatorname{RO})_r(\operatorname{Alk})_m(\operatorname{Ar})_p]_q[\operatorname{B}] \tag{I},$$

$$SiR_{n}^{l}$$
 (RO) _{3-n} (Alkyl) (II),

or

$$SiR_{n}^{1}(RO)_{3-n}(Alkenyl)$$
 (III),

wherein

B is -SCN, -SH, -C1, -NH₂, -OC(O)CHCH₂, -OC(O)C(CH₃)CH₂ (if
$$q = 1$$
) or -Sw- (if $q = 2$), B being bonded chemically to Alk,

R and R¹ are aliphatic, olefinic, aromatic or arylaromatic radicals having 2-30 carbon atoms, which may optionally be substituted by the following groups: hydroxyl, amino, alkoxide, cyanide, thiocyanide, halogen, sulfonic acid, sulfonic ester, thiol, benzoic acid, benzoic ester, carboxylic acid, carboxylic ester, acrylate, meth-acrylate, organosilane radicals, and wherein R and R¹ may have an identical or different definition or substitution,

n is 0, 1 or 2,

Alk is a divalent unbranched or branched hydrocarbon radical having from 1 to 6 carbon atoms,

m is 0 or 1,

Ar is an aryl radical having from 6 to 12 carbon atoms, which may be substituted by the following groups: hydroxyl, amino, alkoxide, cyanide, thiocyanide, halogen, sulfonic acid, sulfonic ester, thiol, benzoic acid, benzoic ester, carboxylic acid, carboxylic ester, organosilane radicals,

p is 0 or 1, with the proviso that p and n are not simultaneously 0,

q is 1 or 2,

w is a number from 2 to 8,

r is 1, 2 or 3, with the proviso that r + n + m + p = 4,

Alkyl is a monovalent unbranched or branched saturated hydrocarbon radical having from 1 to 20 carbon atoms, and

Alkenyl is a monovalent unbranched or branched unsaturated hydrocarbon radical having from 2 to 20 carbon atoms.

Claim 21. The precipitated silica as claimed in claim 1, wherein the CTAB surface area is $170-220 \text{ m}^2/\text{g}$.

Claim 22. The precipitated silica as claimed in claim 1, wherein the CTAB surface area is $245-300 \text{ m}^2/\text{g}$.

Claim 23. The precipitated silica as claimed in claim 1, wherein the BET surface area is $257-300 \text{ m}^2/\text{g}$.

Claim 24. The precipitated silica as claimed in claim 1, wherein the BET surface area is $190-230 \text{ m}^2/\text{g}$.

Claim 25. The precipitated silica as claimed in claim 1, wherein the DBP number is 207 - 276 g/(100 g).

Claim 26. The precipitated silica as claimed in claim 1, wherein the Sears number V_2 is 10-16 ml/ (5 g).

Claim 27. The precipitated silica as claimed in claim 3, wherein the WK coefficient is ≤ 3.0 .

Claim 28. The precipitated silica as claimed in claim 3, wherein the WK coefficient is ≤ 2.5 .

EVIDENCE APPENDIX

Declaration under 37 CFR 1.132 of Andre Wehmeier, filed June 20, 2008.

Declaration under 37 CFR 1.132 of Andre Wehmeier, filed January 15, 2009.



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FOR: HIGHLY DISPERSIBLE PRECIPITATED SILICA

DECLARATION UNDER 37 C.F.R. § 1.132

COMMISSIONER FOR PATENTS ALEXANDRIA, VIRGINIA 22313

SIR:

- I. Andre Wehmeier , declare and state as follows:
- 1. I am a graduate of FH Muenster and received my diploma degree in the year of 1998. I have been employed by

Evonik Degussa GmbH for 10 years as a chemical

graduated engineer in the field of product development

- 2. I am familiar with the claims, and have read the Office Action mailed April 18, 2008, in the above-identified application.
- 3. The following experiments, named Example A, were conducted under my supervision and/or control. In Example A, precipitated silicas according to Examples 4 and 5

of U.S. Patent 6,180,876 (<u>Uhrlandt et al</u>) were compared to precipitated silicas according to Example 1 of the specification of the above-identified application.

4. General performance standard operating procedure (SOP):

The inventive silica according to Example 1 of the above-identified application was tested in a typical motor-truck tire compound. Moreover, this formula is also relevant for the carcass of all rubber tires.

The formula used for the rubber mixtures is specified in the following Table A. The unit "phr" therein denotes parts by weight relative to 100 parts of the raw rubber used.

Table A: Compound

Formula			
Article designation	phr	Substance	Company
1st step	Basic mixing	ixing	
SMR 10 (degraded to ML4 = 60	001	100 Natural rubber	
to 70)			T. I.S. D. L. C. L. D. Main Communic
CORAX N 121	2	Carbon black	Degussa AU; rrankiun am iviam; Ociniany
Silica (KS)	20		
Si 69	2.0	Si 69 (bis(3-triethoxysilylpropyl)tetrasulfide)	Degussa AG; Frankfurt am Maun; Germany
EDENOR STI GS	3.0	Palmitic-stearic acid; "iodine number 1" stearin	Caldic Deutschland GmbH & Co. KG; 40231 Dusseldort;
			Germany
ZnO; RS RAL 844 C	3.0	ZnO	Arnsperger Chemikalien Gmb11; 50858 Cologne; Germany
Vulkanox 4020 / LG	2.0	N-(1,3-Dimethylbutyl)-N'-phenyl-p-	Rhein Chemie Kheinau GmbH; 08219 Manuncim Kucuzu,
		phenylenediamine (6PPD)	Germany
Vulkanox HS/LG	0.5	Trimethyldihydroquinoline	Rhein Chemie Rheinau GmbH; 68219 Mannheim Kheinau;
			Germany
Naffolen ZD	10.0	Aromatic plasticizer oil	Chemetall GmbH; 60487 Frankfurt a. Main; Germany
2nd sten	Lasting	Lasting / remill step	
Sten 1 hatch			
CORAX N 121	2	Carbon black	Degussa AG; Frankfurt am Main; Germany
3rd step	Finish mixing	nixing	
Sten 2 hatch			The state of the s
00 0000	-	80% polymer bound of butylbenzothiazole	Rhein Chemie Rheinau GmbH; 68219 Mannneim Kneinau;
Khenogran 1BBS-80	:	sulfonamide (TBBS)	Germany Certain Desirant
Vulkacit DZ/EG-C	0.3	Dicyclohexylbenzothiazole sulfonamide (DCBS)	Rhein Chemic Rheinau GmbH; 08219 Manufelli Auchlau, Germany
		יייי אין אייי טיי	March VGs 4 · 64771 Darmetadt: Germany
Ground sulfur	1.4	Finely divided sulfur according to rn Eur, Dr	Melch Mondy, of the comment of the c

5. The general method for manufacture of rubber mixtures and their vulcanized derivatives is described in the following book: "Rubber Technology Handbook", W. Hofmann, Hanser Verlag 1994. The specific mixing conditions for the various compounds are presented in Table B.

Table B: Mixing SOP

Mixing SOP	
1" step	W&P GK 1.5E kneader, filling level 0.56,
<u>-</u>	80 rpm, flow temperature 90 °C,
	plunger pressure 5.5 bar
0.0 to 1.0 minutes	Polymers
1.0 to 2.0 minutes	Carbon black; ZnO; stearic acid, Naftolen
2.0 to 3.0 minutes	Silica & silane; other constituents of the 1st step
3.0 to 3.0 minutes	Clean
3.0 to 5.0 minutes	Mix, with speed variation if necessary,
	in order to reach the ejection temperature
5.0 minutes	Discharge batch (batch temperature 140 °C to 150 °C)
	and distribute on roll:
	Cut in and fold over 3 x on left, 3 x on right, turn over
	3 x for narrow roll nip, 3 x for broad roll nip
	Draw out a rolled sheet
24 hours intermediate storage at	room temperature to step 2
2 nd step	W&P GK 1.5E kneader, filling level 0.55,
•	80 rpm, flow temperature 80 °C,
	plunger pressure 5.5 bar
0.0 to 1.0 minutes	Plasticize batch from step 1
1.0 to 2.0 minutes	Carbon black
2.0 minutes	Aerate, clean
2.0 to 4.0 minutes	Maintain batch temperature at 145 ° by speed variation
4.0 minutes	Discharge batch (batch temperature 145 °C to 155 °C)
	and distribute on roll:
	Cut in and fold over 3 x on left, 3 x on right, turn over
	3 x for narrow roll nip, 3 x for broad roll nip
	Draw out a rolled sheet
4 hours intermediate storage at	room temperature to step 3
3 rd step	W&P GK 1.5E kneader, filling level 0.53,
•	40 rpm, flow temperature 50 °C,
	plunger pressure 5.5 bar
0.0 to 2.0 minutes	Batch from step 2, accelerator, sulfur
2.0 minutes	Discharge batch (batch temperature 90 °C to 110 °C)
	and distribute on roll:
	Cut in and fold over 3 x on left, 3 x on right, turn over
	3 x for narrow roll nip, 3 x for broad roll nip
	Draw out a rolled sheet
12 hours intermediate storage a	t room temperature until
beginning of the tests	

6. Technological rubber testing takes place according to the test methods presented in

Table C.

Table C: Test methods

Physical testing	Standard / Conditions	
ML 1+4, 100 °C, 3 rd step (ME)	DIN 53523/3 ISO 667	
Mooney scorch, 130 °C	DIN 53523/3 ISO 667	
Scorch time t ₅ (minutes)		
Scorch time t ₃₅ (minutes)		
Tensile test on standard bar S 1, 23 °C	DIN 53504, ISO 37	
Tensile test (MPa)		
Elongation at break (%)		
DIE C N/mm)	ASTM D 624	
Shore A hardness, 23 °C (SH)	DIN 53 505	
Viscoelastic properties	DIN 53 513, ISO 2856 50 N preliminary force and 25 N amplitude force, temperature-stabilization time 5 minutes; recording of measured values after 30 s test time	
Complex modulus E* (MPa) Loss factor tan δ (-)		

7. Table D below presents the application-related data of the mixtures compounded and tested according to Tables A to C.

Table D: Results

		Example 4 Uhrlandt et al	Example 5 <u>Uhrlandt et al</u>	Example 1 of present invention
ML 1+4, 100 °C, 3 rd step	ME	37	39	39
Mooney scorch 130 °C; small rotor				
Scorch time t ₅	min	17.9	18.1	19.9
Scorch time t ₃₅	min	22.2	22.7	23.9
Vulcanization time; 150 °C	min	18	18	18
Tensile strength	MPa	22.7	22.3	22.7
Elongation at break	%	577	563	577
Die C; 100 °C	N/mm	52	55	75
Shore A hardness	SH	56	55	55
Viscoelastic properties				
E*,0 °C	MPa	8.0	7.9	8.1
E*, 60 °C	MPa	5.6	5.6	5.6
tan δ, 0 °C	-	0.234	0.234	0.234
tan δ, 60 °C		0.132	0.137	0.139

Application No. 10/523,029 Declaration Under 37 C.F.R. § 1.132

8. The compound containing the silica according to Example 1 of the present

invention exhibits a very balanced profile of rubber values; among the properties of the raw

mixture, the improved processing safety stands out in particular, as indicated by the

prolonged scorch times. Whereas the reinforcement properties and also the viscoelastic

properties correspond to the reference level or are slightly improved at comparable Shore A

hardness, a distinct increase of high-temperature tearing resistance in the ASTM D 624 test

(Die C) can be observed. This performance improvement by 36% and 44% respectively is an

important criterion above all for high-performance, SUV and motor-truck tires.

9. The undersigned declares further that all statements made herein of his own

knowledge are true and that all statements made on information and belief are believed to be

true; and further that these statements were made with the knowledge that willful false

statements and the like so made are punishable by fine or imprisonment, or both, under

Section 1001 of Title 18 of the United States Code and that such willful false statements may

jeopardize the validity of this application or any patent issuing thereon.

10. Further declarant saith not.

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27.05.200 P

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FOR: HIGHLY DISPERSIBLE

PRECIPITATED SILICA

SECOND DECLARATION UNDER 37 C.F.R. § 1.132

COMMISSIONER FOR PATENTS ALEXANDRIA, VIRGINIA 22313

SIR:

- I, André Wehmeier, declare and state as follows:
- 1. I am the same André Wehmeier who executed a Declaration under 37 C.F.R. § 1.132, with an execution date of May 27, 2008 (first Wehmeier Declaration) in the above-identified application.
- 2. I am familiar with the claims, and have read the Office Action mailed July 31, 2008, in the above-identified application.
 - 3. The following experiments were conducted under my supervision and/or control.
- 4. New Examples 1A and 1B, which are according to the present invention, were prepared as follows:

Example 1A

80 l deionized water were introduced into a 120 l reactor with double shell heating and heated indirectly with steam until a temperature of 83°C was reached. Then, while stirring vigorously (shearing) at 83 °C for 100 minutes, 194.56 g/min water glass (density

1.346 kg/l, 27% SiO₂, 8.00% Na₂O) and approx. 49.77 g/min sulfuric acid (density 1.398 kg/l, 50.1% H₂SO₄) were added. This sulfuric acid was metered in such a way that a pH of 8.5 (measured at room temperature) prevails in the reaction medium. The reaction heat occurring during the reaction was dissipated via external cooling in order to maintain the reaction temperature.

After completion of the precipitation the addition of water glass was stopped and the sulfuric acid was added at 49.77 g/min until a pH of 3.0 (measured at room temperature (20°C)) was achieved.

The suspension that was obtained in this manner was filtered using a membrane filter press and rinsed with water. The filter cake was dried using a spin flash dryer.

The pulverulent product that was thus obtained had a BET surface area of 198 m²/g, a CTAB surface area of 193 m²/g, DBP absorption of 252 g/(100 g), and a Sears number V_2 of 13.5 ml/(5 g).

Example 1B

80 l deionized water were introduced into a 120 l reactor with double shell heating and heated indirectly with steam until a temperature of 68°C was reached. Then, while stirring vigorously (shearing) at 68°C for 100 minutes, 194.56 g/min water glass (density 1.346 kg/l, 27% SiO₂, 8.00 % Na₂O) and approx. 49.77 g/min sulfuric acid (density 1.398 kg/l, 50.1% H₂SO₄) were added. This sulfuric acid was metered in such a way that a pH of 8.5 (measured at room temperature) prevails in the reaction medium. The reaction heat occurring during the reaction was dissipated via external cooling in order to maintain the reaction temperature.

After completion of the precipitation the addition of water glass was stopped and the sulfuric acid was added at 49.77 g/min until a pH of 3.0 (measured at room temperature (20°C)) was achieved.

The suspension that was obtained in this manner was filtered using a membrane filter press and rinsed with water. The filter cake was dried using a spin flash dryer.

The pulverulent product that was thus obtained had a BET surface area of 272 m²/g, a CTAB surface area of 245 m²/g, DBP absorption of 243 g/(100 g), and a Sears number V_2 of 18.3 ml/(5 g).

- 5. As can be seen above, new Example 1A has a BET surface close to the lower limit of presently-pending Claim 1 of the above-identified application. The Sears number and the CTAB value are also close to the lower limit of the corresponding ranges of said Claim 1. New Example 1B, in contrast thereto, was designed as representative for a silica close to the upper limits of said Claim 1. BET, CTAB and Sears number of new Example 1B are close to the upper limits of said Claim 1. The respective values for BET, CTAB and Sears number as described in the specification herein for Example 1 are in between the two new Examples 1A and 1B.
- 6. The BET surface area, CTAB surface area, DBP number and Sears number for new Examples 1A and 1B, and Example 4 of U.S. 6,180,876 (Uhrlandt et al) which, in my opinion, is the silica disclosed in the prior art that is closest to the invention of presently-pending Claim 1, are shown in the following Table C:

Table C

		Example 4	New Example	New Example
		Uhrlandt et al	1A	1B
BET surface area	m²/g	178	198	272
CTAB surface area	m^2/g	156	193	245
DBP number	g/(100g)	282	252	243
Sears number V2	ml/(5g)	18.7	13.5	18.3

- 7. The silicas prepared by Examples 1A and 1B were then evaluated according to the same procedures as described in paragraphs 4 and 5 of the first Wehmeier Declaration, which paragraphs are hereby incorporated by reference.
- 8. Die C; 100°C (N/mm) for Examples 1A and 1B were measured according to paragraph 6 of the first Wehmeier Declaration, which paragraph is hereby incorporated by reference.
- 9. The high-temperature tearing resistance test was performed according to ASTM D 624 test (Die C). The test results are shown in following Table D:

Table D

		Example 4	New Example	New Example
		<u>Uhrlandt et al</u>	IA.	IB
Median Die C	N/mm	64	67	91
(ASTM D 624)				
9 single values (100°C)				

10. The comparison tests revealed that new Example 1A with

$$BET = 198 \text{ m}^2/\text{g}$$

$$CTAB = 193 \text{ m}^2/\text{g}$$

Sears number =13.5 ml/mg

shows the worst Median Die C value of all examples of the present invention. This Die C value, however, is still better than that of the closest prior art, i.e. Example 4 of <u>Uhrlandt at al</u>.

With increasing BET, CTAB and Sears number a trend can be seen. Accordingly, new Example 1B, which has the highest values for BET, CTAB and Sears number consequently shows the highest Die C value of 91, which is more than 42% better than Example 4 of <u>Uhrlandt et al.</u>

- 11. Besides a very balanced profile of rubber values, both new Examples 1A and 1B show that also in that very broad specific surface area range the improvement of high-temperature tearing resistance is given. This is of most interest for all high-performance, SUV, truck and also for motor bike tires in the tread compound as well as in all carcass compounds.
- 12. All three examples of the invention (Example 1 and new Examples 1A and 1B) show an improved high temperature tear resistance compared to <u>Uhrlandt et al</u>. Those silicas represent the whole range now claimed in the claims of the above-identified application and therefore prove, that the presently-claimed invention shows significant improvements compared to the closest prior art.
- 13. The Die C value for Example 4 of <u>Uhrlandt et al</u> was found to be 52 N/mm, under the experiment conducted for and as shown in the first Wehmeier Declaration, while the corresponding value herein, as shown in paragraph 9, was found to be 64 N/mm. The reason for the discrepancy is explained as follows.
- 14. Even if the same rubber is used, there are differences between the properties of different rubber lots. In addition, unavoidable variations of the mixing conditions have an influence on the physico-chemical properties of the rubber mixture. Even if the recipe is the same, variations of the test results are not unusual. Consequently, only data of one and the same test series can be compared directly and absolutely. In the above-described experiments of this Declaration, the median Die C value based on nine individual tests was used as the Die C value. In the first Wehmeier Declaration, only three single tests were conducted. For the above-described experiments of this Declaration, a new rework of Example 4 of <u>Uhrlandt et al</u> was used. It is not unusual that there is some variation between different reworks. As explained above, some variations of absolute values of Die C are usual between different test series. Nevertheless, within one test series, such as shown in the first Wehmeier Declaration,

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the comparison of Die C values is still valid. Thus, even though the absolute values for Die C

for Example 4 of Uhrlandt et al are different between this Declaration and the first Wehmeier

Declaration, nevertheless, the data validly show improved Die C values compared to

Uhrlandt et al.

15. As explained above, the absolute Die C value depends on the rubber mixture as

well as the mixing conditions. Since different rubber mixtures are used for truck tires and bus

tires, a minimum value of Die C cannot be defined.

16. The undersigned declarant declares further that all statements made herein of his

own knowledge are true and that all statements made on information and belief are believed

to be true; and further that these statements were made with the knowledge that willful false

statements and the like so made are punishable by fine or imprisonment, or both, under

Section 1001 of Title 18 of the United States Code and that such willful false statements may

jeopardize the validity of this application or any patent issuing thereon.

17. Further declarant saith not.

Signature

Customer Number

22850

Tel. (703) 413-3000 Fax. (703) 413-2220 (OSMMN 05/06) Date

RELATED PROCEEDINGS APPENDIX

None.